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Procedia Engineering 47 (2012) 302 – 305

Procedia Engineering

www.elsevier.com/locate/procedia

Proc. Eurosensors XXVI, September 9-12, 2012, Kraków, Poland

Effect of photoresist coating on the reusable resonant cantilever sensors for assessing exposure to airborne nanoparticles

Hutomo Suryo Wasisto[1]* , Stephan Merzsch[1], Andreas Waag[1], Ina Kirsch[2], Erik Uhde[2], Tunga Salthammer[2], and Erwin Peiner[1]

[1]*Institute of Semiconductor Technology, TU Braunschweig, Hans-Sommer Str. 66, Braunschweig D-38106, Germany*
[2]*Material Analysis and Indoor Chemistry Department, Fraunhofer WKI, Bienroder Weg 54E, Braunschweig D-38108, Germany*

Abstract

Silicon cantilever resonators were designed and fabricated in various shapes and dimensions to be used in an airborne nanoparticle (NP) sensing application. For a repeatedly usable sensor, the cantilever surface was covered by a sacrificial layer of photoresist prior to the airborne NP sampling. Using the electrostatic precipitation method, airborne NPs were sampled on the cantilever. Within a lift-off process of wet cleaning, the photoresist layer was removed together with the trapped NPs. The resonant frequency and the quality factor (*Q*-factor) of the fundamental and higher order flexural modes of the silicon cantilevers were characterized for different thickness given by viscosity of the photoresist layer. It was found that the proposed recycling technique was most effective when a thin photoresist film was used. By using higher order resonant modes, *Q*-factors of more than 1000 in air were maintained even after the photoresist coating.

Keywords: cantilever resonators; photoresist; airborne nanoparticles; quality factor

1. Introduction

Recently many modern applications are manufactured using nanoparticles (NPs). Despite the NP benefits, there is increasing attention to the potential health risk of NPs released to the ambient especially

* Corresponding author. Tel.: +49-531-391-3776; fax: +49-531-391-5844.
E-mail address: h.wasisto@tu-bs.de.

doi:10.1016/j.proeng.2012.09.143

at workplaces [1]. For monitoring of personal exposure to airborne NPs, MEMS resonant sensors have been proposed based on a FBAR [2] or a silicon cantilever [3] as in the present work. However, research on a cleaning method for recycling of the polluted airborne NP sensors was still rare. In this work a photoresist film is used as a coating layer for sensor recycling purpose. The effects of photoresist on the resonant frequency and the *Q*-factor of the silicon cantilever are also investigated.

2. Effect of Photoresist Coating on the Silicon Cantilever Sensor

The silicon cantilever sensors integrated with full piezoresistive Wheatstone bridges were fabricated using a silicon bulk micromachining process. The details of sensor fabrication were already described in detail previously [3, 4]. As intended to be used in a handheld detection system of airborne NP based on the resonant frequency shift induced by trapped NP mass, the sensor was designed to have high mass sensitivity and high *Q*-factor. Moreover, a miniaturized and lightweight electrostatic NP sampler was combined into the system [5]. Since the cantilever gets fully covered by NPs over the continuous NP sampling, the operating life of a sensor is limited. To extend this time a sensor recycling method by covering the silicon cantilever with a sacrificial layer was proposed and investigated. In this process, particles settle on top of the layer instead of the silicon. Within a wet cleaning, not only the additional layer is removed, but also most of the trapped NPs as illustrated in Fig. 1.

In the first experiment, a bare cantilever with hexagonal shaped NP sampling area at its free end was immersed into a solution of AZ 5214E and its thinner PGMEA mixed to a composition of 1 : 1 (Fig. 2(a)). Consequently, the *Q*-factors of the resonator had been reduced by more than an order of magnitude when measured in air (Fig. 2(b)). At the sixth resonant mode, the *Q*-factor of the bare cantilever Q_{hex_bare6} = 3400 dropped to $Q_{hex\_coated6}$ = 135 attributed to the additional surface stress induced by a relatively thick and heavy photoresist layer.

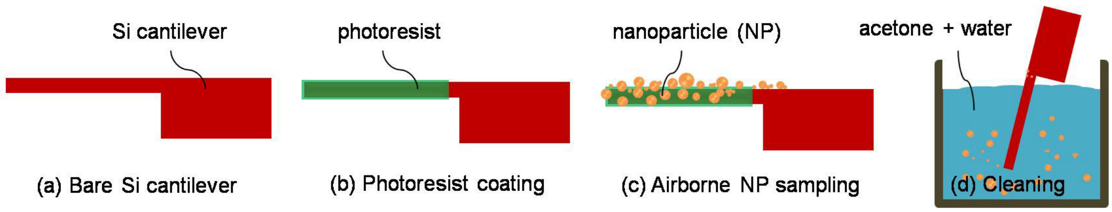

Fig. 1. Cycle of silicon cantilever sensor during its use started from (a) bare condition, (b) photoresist coating, (c) airborne NP sampling, and (d) lift-off by ultrasonic agitation.

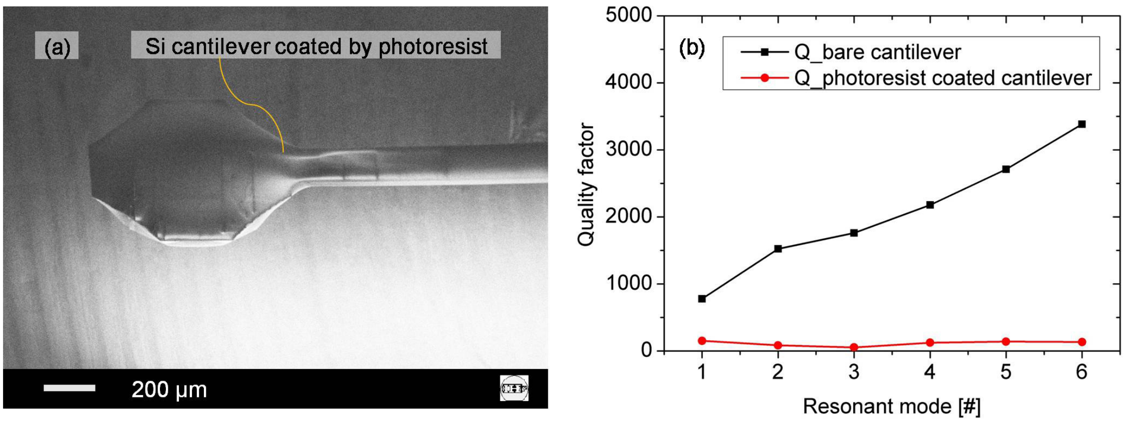

Fig. 2. (a) Silicon cantilever with hexagonal-shaped free end coated by thick photoresist and (b) its measured *Q*-factors of the first six resonant modes before and after photoresist coating.

To avoid this not tolerable drop of *Q*-factors, it is favorable to use a thin and lightweight photoresist layer. Thus, the photoresist-thinner density ratio was reduced to 1 : 9 in the second experiment using a slender silicon cantilever ($2750 \times 100 \times 50\ \mu m^3$). After immersion and hardbake, the resonant frequencies and *Q*-factors of the first three flexural modes were measured as operating in higher resonant modes can increase mass sensitivity of the cantilever sensor [6]. At the second and third modes, the *Q*-factors were still above 1200 (Fig. 3(a)).

3. Detection of Airborne Nanoparticle

After investigation of photoresist thickness dependence, the sensor was assessed in an airborne carbon NP ambient under typical workplace conditions (V = 1 m^3, T = 23 °C, rH = 30 %, p = 1 atm). A concentration of ~38600 NP/cm³ was maintained by constant output atomizer (TSI 3076) for 15 min. During NP sampling, the NPs size distribution was monitored by a fast mobility particle sizer (FMPS, TSI 3091) at 1 Hz time resolution. At an aerosol flow rate through the sampler of 680 cm^3/min and a carbon density of 2.27 g/cm^3, a total number of flowing NPs of 3.94 x 10^8 particles and the total mass of flowing NPs of 689.2 ng are found assuming that the NPs have a spherical shape. As shown in Fig. 3(b), the resonant frequency shifts after the deposition of carbon NPs on the photoresist-coated cantilever.

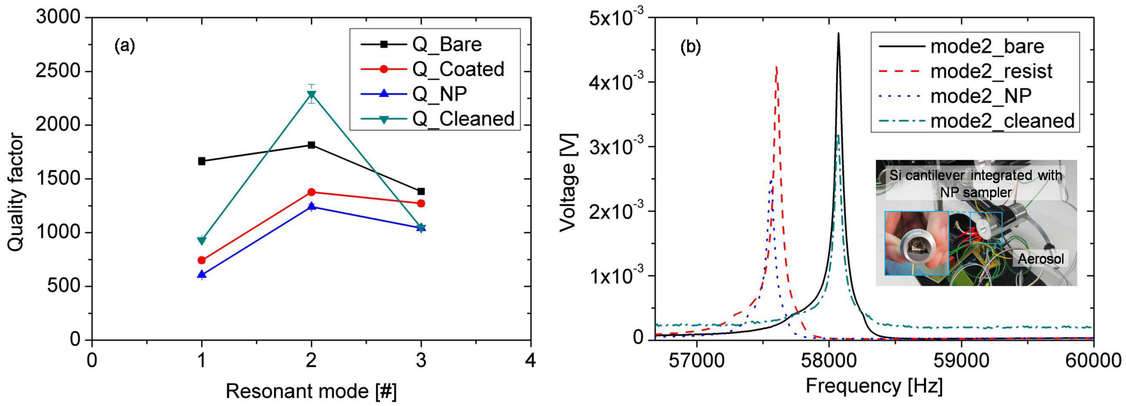

Fig. 3. (a) Measured *Q*-factors of the first three resonant modes of slender silicon cantilever during recycling process; (b) resonant frequency shifts of the second resonant mode (inset: Si cantilever under airborne NP sampling).

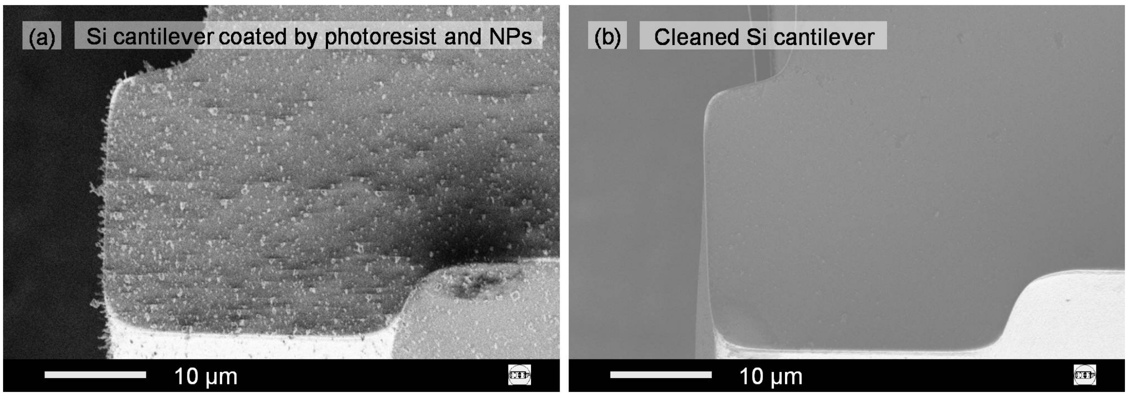

Fig. 4. SEM images of Si cantilever in (a) coated state and (b) cleaned state after recycling process.

The surface condition of the cantilever free end after NP sampling is depicted in Fig. 4(a) showing uniform deposition of carbon NPs on a photoresist-coated cantilever. Under assumption that photoresist layer does not give any effects on cantilever stiffness and the sensor was operated in the second resonant mode, total masses of resist covering the sensor $m_{resist} = 2 \times m_{cant} \times \Delta f_{resist} / f_{cant2} = 516.91 \pm 0.18$ ng and combined carbon NPs with resist $m_{resist+NP} = 2 \times m_{cant} \times \Delta f_{resist+NP} / f_{cant2} = 561.02 \pm 0.18$ ng can be calculated with a bare cantilever weight $m_{cant} = 32.04$ µg, a bare resonant frequency $f_{cant2} = 58068.78 \pm 0.17$ Hz, a resonant frequency shift after resist coating $\Delta f_{resist} = 468.46 \pm 0.12$ Hz, a resonant frequency shift after NP sampling $\Delta f_{resist+NP} = 508.43 \pm 0.12$ Hz. Finally, the mass of collected NPs can be estimated to be $m_{NP} = m_{resist+NP} - m_{resist} = \sim 44.11 \pm 0.18$ ng. Furthermore, the mass sensitivity of the sensor $S = 0.91$ Hz/ng is calculated. The sampling efficiency can also be analyzed by comparing to the total mass of flowing NPs with the mass of collected NPs which yields 6.4 %. After immersing the cantilever in a solution of acetone and deionized water inside an ultrasonic bath, the cantilever was cleaned (Fig. 4(b)) and the resonant frequency of the sensor has been shifted back to the first condition, i.e. bare condition. In terms of the resonant frequencies of the cantilever before and after recycling process, a cleaning efficiency of 99.68 % has been achieved.

4. Conclusion

A method of recycling a cantilever sensor after being polluted by airborne NPs has been tested using a sacrificial layer, i.e. photoresist. The effects of the photoresist coating were studied. By means of the experimental results, a thin and lightweight photoresist (photoresist-thinner density ratio = 1 : 9) layer has less drops of Q-factors of the resonant sensor which shows the feasibility of using this method in order to extend the operating life of the sensor.

Acknowledgements

The authors thank S. Wientzek, J. Arens, and D. Rümmler for their valuable technical assistances. This work is supported by the German Federal Ministry of Education and Research (BMBF) in the collaborative project “NanoExpo” within the cluster “NanoCare” under no. 03X0098A.

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